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The effects of pyrolytic conditions on microwave pyrolysis of prairie cordgrass and kinetics

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ABSTRACT

This study investigated microwave pyrolysis of prairie cordgrass (PCG) to determine the effects of pyrolysis temperature and time on the yields of bio-oil, syngas, and biochar. Microwave pyrolysis of PCG was analyzed using response surface methodology (RSM) to find out the effect of process variables on the bio-oil and syngas yield and established prediction models. Second-order reaction kinetics was developed to model the PCG pyrolysis. Bio-oil recovery was in the range of 20.3–33.1 wt% of the biomass. GC–MS analysis indicates that the bio-oil contained a series of important and useful chemical compounds: aliphatic hydrocarbons and aromatic hydrocarbons. These chemical compounds evolved were related to the pyrolysis conditions.

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1. Introduction

Prairie cordgrass (PCG) is a native perennial grass that grows widely across the Great Plains and is found throughout the northern United States and southern Canada [1–3]. PCG has a reasonable yield of 10 tons of dry matter/ha which makes it a renewable energy source for power generation and residential heating [4]. Boe and Lee demonstrated that PCG has the most northerly distribution of any of the C4 perennial grasses, which may give it a wider environmental range than switchgrass for biomass production in the northern Great Plains [4].

Since the 1970s energy crisis the production of renewable fuels and chemicals from lignocellulose biomass has been extensively studied. Comparing with fossil fuels, it is seen that biomass generates the same amount of CO_2 as fossil fuels per unit C, but every time a new plant grows, it removes the same amount of CO_2 from the atmosphere [5].

According to McKendy [6], thermochemical conversions of lignocellulose biomass (i.e., combustion, pyrolysis, or gasification) are receiving increasing interest and utility as a method for production of renewable energy and fuels because it offers numerous advantages.

Microwave pyrolysis is one of the most efficient thermochemical ways of converting biomass into high value products such as bio-oil, syngas, biochar, and chemicals. Microwave pyrolysis overcomes some disadvantages of conventional pyrolysis methods such as slow heating and necessity of feedstock shredding. It also provides some advantages including increased product yield, environmental compatibility, process time and energy saving, and low requirements for space and capital equipment [7]. The microwave-assisted pyrolysis method has been successfully applied to plant residues [8,9], automotive industry and plastic wastes [10,11], wood [12,13] and sewage sludge [14]. It is reported that material properties and operating conditions are the main factors affecting microwave pyrolysis process, since they determine the characteristics and yields of the final products [15]. Previous study also showed temperature and pyrolysis time were the important factors in the microwave pyrolysis of corn stover [9] and distils dried grain with soluble [16]. But microwave pyrolysis of prairie cordgrass has not been found in literature.

Understanding the chemical composition during the pyrolysis is a key feature in determining potential uses and values of biomass resources. Pyrolysis of the biomass constitutes a viable source of more than 230 organic chemicals [17]. The knowledge of pyrolysis products can also facilitate the research on the cheap chemicals and char for the preparation of carbon molecular sieves [18].

The objective of this study was to investigate microwave pyrolysis of PCG and determine the effects of pyrolytic conditions on the yields of bio-oil, syngas, and biochar. The relationship between product yield and pyrolysis conditions was investigated and kinetics models were established to predict product yields. GC–MS

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Table 1
Experimental design and results for microwave pyrolysis of PCG.

Std ID	Reaction temperature (°C)	Residence time (min)	Oil weight (g)	Gas weight (g)	Biochar weight (g)	Volatile weight (g)
1	550	8	27.7	41.8	30.5	69.5
2	650	8	28.0	45.3	26.7	73.3
3	550	18	30.2	42.8	27.0	73.0
4	650	18	33.1	48.4	18.5	81.5
5	529.3	13	20.3	32.0	47.7	52.3
6	670.7	13	31.4	45.0	23.6	76.4
7	600	5.9	23.6	43.9	32.5	67.5
8	600	20.1	32.5	53.7	13.8	86.2
9	600	13	24.2	46.3	29.5	70.5
10	600	13	24.3	45.5	30.2	69.8
11	600	13	24.4	47.9	27.7	72.3
12	600	13	24.3	46.5	29.2	70.8
13	600	13	24.1	47.1	28.8	71.2

methods were employed in this investigation to understand the chemical composition of bio-oils.

2. Materials and methods

2.1. Materials

PCG was obtained from the Department of Plant Science, South Dakota State University. These biomasses were grounded to produce pieces using a 4-mm sieve (Speedy King, Winona Attrition Mill Co., MN, USA). Grounded samples were dried in oven at 80 °C for 24 h and kept in sealed plastic bags at room temperature (25 °C) prior to microwave pyrolysis.

2.2. Microwave pyrolysis apparatus

A Sineo MAS-II batch microwave oven (2450 MHz, Shanghai Microwave Chemistry Technology Co. Ltd., China) with a rated power of 1000 W was used to convert PCG to bio-oil at the 700 W power setting. This power setting gave a heating rate of 94.5 °C/min before desired temperatures. 100 g biomass was fed into a 3-neck flask. One neck of the flask was connected to nitrogen that offered an air-free environment in the pyrolysis process. The second neck was connected with a set of condensers. In the process, volatiles were passed through a series of condensers in a coldwater where the water and condensable bio-oil components were condensed while non-condensable volatiles escaped as syngas at the end of the condensers. The third neck of the flask was connected to a temperature sensor (Shanghai Microwave Chemistry Technology Co. Ltd., China) in order to investigate the effect of the pyrolysis temperature (Fig. 1).

The system was purged with nitrogen for 15 min prior to microwave pyrolysis to create an oxygen-free gas environment. At the end of the experiments, the oil fraction was collected from the condensers and weighed. The solid char was removed and weighed. Syngas yield was determined by overall material balance:

$$\text{weight}_{\text{syngas}} = 100\,\text{g} - \text{weight}_{\text{bio-oil}} - \text{weight}_{\text{biochar}} \quad (1)$$

2.3. GC–MS analysis

The oil product was analyzed by GC–MS using a Varian Star 3400 CX gas chromatograph coupled to a Varian Saturn 2000 detector (Varian Inc., USA). The gas chromatograph was equipped with a 10 m × 0.25 mm ID, 0.5 μm film thickness RTX-5Sil MS capillary column (Restek, Bellefonte, PA, USA). Helium was employed as carrier gas at a constant flow rate of 0.6 ml/min. The initial temperature of oven was 45 °C held for 2 min and then programmed from 45 °C to 300 °C at 10 °C/min with an isothermal held for 10 min. The injector temperature was 300 °C and the injection size was 1 μl. The ion source temperature was 230 °C for the mass selective detector. The compounds were identified by comparing data with NIST98 Mess Spectral Library search.

2.4. Experimental design

Two factors, pyrolysis temperature (X_1, °C) and residence time (X_2, min), were selected as the independent variables, and a central composite design (CCD) was performed according to Liu et al.'s method [19]. The reaction time was recorded after the desired temperature was reached. The yields of bio-oil, syngas and biochar (Y_i, %) was used as dependent output variables.

The central values and ranges were the following: central pyrolysis temperature 600 °C, 529–671 °C range and central residence time 13 min, 5.9–20.1 min range (Table 1). The experimental results were fitted using a polynomial quadratic equation (Eq. (2)) in order to correlate the response variables to the independent variables.

$$Y_i = b_0 + b_1X_1 + b_2X_2 + b_{11}X_1^2 + b_{22}X_2^2 + b_{12}X_1X_2 \quad (2)$$

where Y_i is predicted response, b_0 is offset term, b_1 and b_2 are liner effects, b_{11} and b_{22} are squared effects, and b_{12} is interaction term.

2.5. Pyrolysis kinetics

A kinetic scheme to model thermal decomposition of biomass can be schematized as:

$$\text{PCG} \rightarrow \text{char} + \text{volatiles} \quad (3)$$

where the reaction rate is a function of the remaining raw material and follows an Arrhenius law dependent on temperature [20–22].

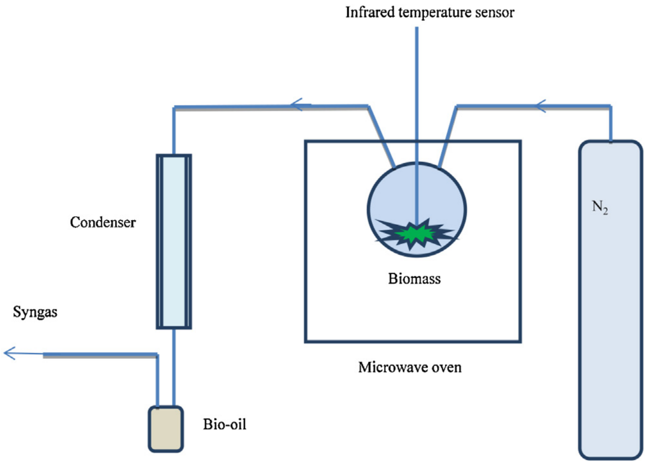

Fig. 1. Schematic of microwave pyrolysis apparatus.

The rate of decomposition can be expressed by the following equation:

$$\frac{d\alpha}{dt} = A\exp\left(-\frac{E}{RT}\right)(1-\alpha)^n \tag{4}$$

where A is the frequency or pre-exponential factor of the pyrolysis process ($s-1$), E is the apparent activation energy (J/mol), T is the temperature (K), R is the universal gas constant, 8.3145 (J/mol K), n is the order of reaction, t is the time (s), and the fractional reaction α is defined in terms of the change in mass of the SG sample:

$$\alpha = \frac{X_0 - X}{X_0 - X_f} \tag{5}$$

where X_0 is the initial weight, X is the actual weight after each condition of the pyrolysis tests, and X_f is the final weight of char which cannot be pyrolyzed.

The Coasts–Redfern method was used to solve Eq. (4) to determine the values of the kinetic parameters.

2.6. Coast–Redfern method

For a constant heating rate β ($\beta = dT/dt$), Eq. (4) can be expressed as

$$\frac{d\alpha}{dT} = \frac{A}{\beta}e^{-E/RT}(1-\alpha)^n \tag{6}$$

Eq. (6) can be rearranged and integrated as

$$\int_0^{\alpha}\frac{d\alpha}{(1-\alpha)^n} = \frac{A}{\beta}\int_0^{T} e^{-E/RT}\,dT \tag{7}$$

Integrating $1/(1-\alpha)^n$ and $e^{-E/RT}$ in Eq. (7), the following expression can be obtained:

$$\frac{1-(1-\alpha)^{1-n}}{1-n} = \frac{ART^2}{\beta E}e^{-E/RT}\left(1-\frac{2RT}{E}\right) \tag{8}$$

Transforming Eq. (8) into a logarithmic expression:

$$\ln\left(\frac{1}{T^2}\cdot\frac{1-(1-\alpha)^{1-n}}{1-n}\right) = \ln\frac{AR}{\beta E}\left(1-\frac{2RT}{E}\right) - \frac{E}{R}\cdot\frac{1}{T} \tag{9}$$

Owning to $(2RT/E) \ll 1$, $2RT/E$ can be neglected. This makes $1-(2RT/E) \approx 1$. Eq. (9) becomes Eq. (10) when $n \neq 1$ and Eq. (11) when $n = 1$:

$$\ln\left(\frac{1}{T^2}\cdot\frac{1-(1-\alpha)^{1-n}}{1-n}\right) = \ln\frac{AR}{\beta E} - \frac{E}{R}\cdot\frac{1}{T} \quad n \neq 1 \tag{10}$$

$$\ln\left(\frac{-\ln(1-\alpha)}{T^2}\right) = \ln\frac{AR}{\beta E} - \frac{E}{R}\cdot\frac{1}{T} \quad n = 1 \tag{11}$$

Thus, a plot of $\ln((1/T^2)\cdot(1-(1-\alpha)^{1-n})/(1-n))$ versus $1/T$ when $n \neq 1$ or $\ln(-\ln(1-\alpha)/T^2)$ versus $1/T$ when $n = 1$ should result in a straight line with slop $= -E/R$ and intercept $= \ln(AR/\beta E)$.

3. Results and discussion

3.1. Effects of reaction temperature and time on yields of bio-oil, syngas and volatile

The experimental volatile, syngas, and bio-oil yield is given in Table 1. The yield range of volatile was found from 52.3 to 86.2% varied with different pyrolysis conditions, bio-oil was from 20.3 to 33.1%, and syngas was from 32.0 to 53.7%. The yields are calculated based on weight.

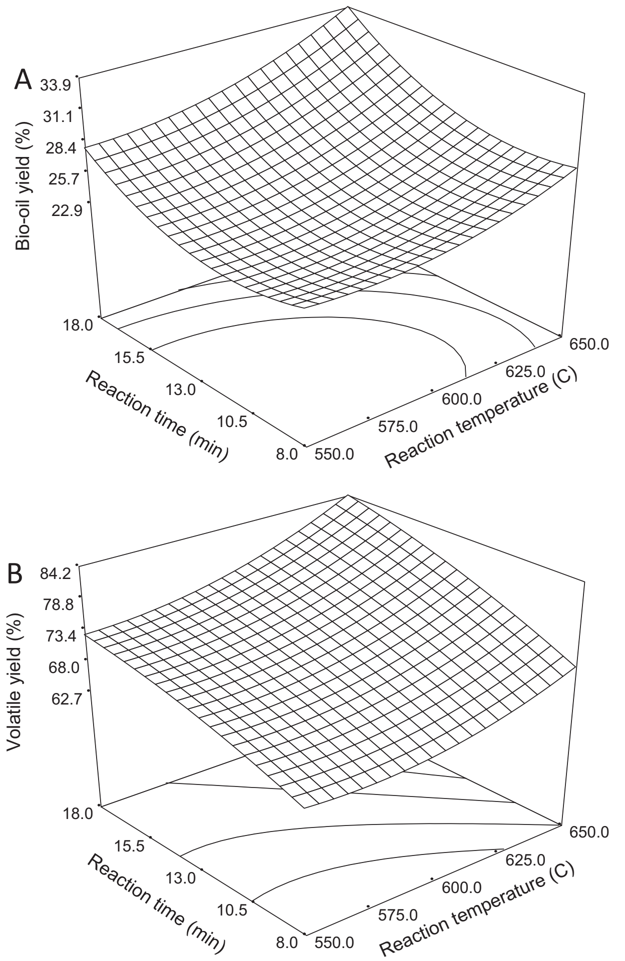

Fig. 2. The response surface plots based on bio-oil (A) and volatile yield (B) as a function of pyrolysis time and temperature.

The results from the experiment were modeled using a second-order, multifactor regression equation. Thirteen experiments were performed using different combinations of the variables as per the CCD. The experimental design matrix and yields of products by microwave pyrolysis are shown in Table 1. Eq. (2) was reduced by using backward statistical analysis, and parameters were sequentially removed based on the coefficient's p-value until all remaining were significant ($p < 0.05$). Using the results of experiments, second order polynomial regression equations (Eqs. (12)–(14)) were obtained to model the yield of bio-oil, syngas and yolatile.

$$Y_{\text{bio-oil}} = 24.26 + 2.52X_1 + 2.36X_2 + 2.59X_1^2 \tag{12}$$

$$Y_{\text{syngas}} = 46.66 + 2.24X_1 + 3.44X_2 - 3.85X_2^2 \tag{13}$$

$$Y_{\text{volatile}} = 70.92 + 24.77X_1 + 5.76X_2 + 3.88X_1^2 \tag{14}$$

where Y presents the yield of bio-oil, syngas and volatile and X_1 and X_2 are pyrolysis temperature and residence time respectively.

The correlation coefficients of determination, R^2 were 0.8, 0.9 and 0.84, respectively for bio-oil, syngas and volatile, implies that the reduced regression models can be used to explain the pyrolysis reaction. The small p-values (<0.05) confirmed that both reaction temperature and residence time significantly influenced the yield of bio-oil, syngas and volatiles. The interaction effect was not significant on the yield of bio-oil, syngas and volatiles between reaction temperature and residential time.

3.2. Response surface analysis

Fig. 2 illustrates a typical response surface for bio-oil (A) and volatile (B) production from PCG. Both bio-oil yield and volatile yield increased with the increase of reaction temperature and

Table 2
Major chemical composition of bio-oil from microwave pyrolysis of PCG at different conditions: PCG1: 550 °C and 8 min; PCG2: 550 °C and 18 min; PCG3: 650 °C and 8 min; and PCG4: 650 °C and 18 min.

Time	Name	Formula	PCG1	PCG2	PCG3	PCG4
3.002	Acetic acid, anhydride with formic acid	$C_3H_4O_3$	0.362	0.178	0.223	0.485
3.153	Pyridine, 3-methyl-	C_6H_7N	0.758	0.331	0.740	1.955
3.332	Bicyclo [1,2,2] heptane, 2-ethyl-	C_9H_{16}	9.910	–	11.510	9.610
3.657	2-Cyclopenten-1-one, 2-hydroxy-	$C_5H_6O_2$	8.43	3.762	7.703	8.886
3.826	1,6;2,3-Dianhydro-4-O-acetyl-, beta,-d-talopyranose	$C_8H_{10}O_5$	4.398	1.591	3.811	4.425
4.169	Pentadienal, 2-methyl-	C_6H_8O	1.609	1.222	1.791	1.432
4.479	Cyclopropane, 2-(1,1-dimethyl-2-pentenyl)-	$C_{12}H_{22}$	3.837	2.400	4.467	3.884
4.825	Pentyl glycolate	$C_7H_{14}O_3$	0.466	0.231	0.833	–
4.882	2-Cyclopenten-1-one, 2-hydroxy-	$C_5H_6O_2$	1.265	0.779	1.586	–
5.339	Heptane, 2,3,6-trimethyl-	$C_{10}H_{22}$	0.555	0.294	1.143	–
5.432	2-Furancarboxaldehyde, 5-methyl-	$C_6H_6O_2$	1.757	1.370	0.802	0.781
5.516	Lyratol	$C_{10}H_{16}O$	4.49	2.145	2.003	1.948
5.647	6-Methoxy-4′-methylaurone	$C_{17}H_{14}O_3$	1.495	0.857	0.934	0.968
5.834	Phenol	C_6H_6O	7.324	6.293	6.608	6.017
6.028	6-Methyl-1,5-heptadiene	C_8H_{14}	0.99	–	0.692	0.606
6.159	Heptane, 1-(2-propenyloxy)-	$C_{10}H_{20}O$	–	–	3.289	3.281
6.545	2,3,3-Trimethyl-1-hexene	C_9H_{18}	2.067	–	3.028	–
6.594	2-Ethyl-5-propylcyclopentanone	$C_{10}H_{18}O$	–	1.510	–	2.855
6.752	3-Heptyne, 5-methyl-	C_8H_{14}	–	1.5	–	1.866
6.889	Indene	C_9H_8	–	0.753	–	0.238
7.154	Phenol, 2-methyl-	C_7H_8O	5.751	6.297	5.629	5.999
7.772	Mequinol	$C_7H_8O_2$	10.735	9.492	10.273	11.077
7.803	1,6-Decadiene, 2,6,9-trimethyl-, (E)-	$C_{13}H_{24}$	1.819	1.003	–	–
8.234	4′-Hydroxy-6-methoxyaurone	$C_{16}H_{12}O_4$	1.264	0.984	1.311	1.073
8.682	Phenol, 2,4-dimethyl-	$C_8H_{10}O$	1.89	0.825	0.581	–
8.992	Phenol, 3-ethyl-	$C_8H_{10}O$	1.385	1.472	0.590	1.498
9.167	6-Oxabicyclo[3,2,1]oct-2-ene, 2,8,8-trimethyl-7-propyl-	$C_{13}H_{22}O$	0.515	–	1.583	–
9.286	(−)-Methyl norcaryl ketone	$C_9H_{14}O$	3.887	5.191	4.112	5.600
9.869	Benzaldehyde, 4-methyl-	C_8H_8O	–	4.159	2.019	–
10.020	2-Propenoic acid, 3-(1-cyclopenten-1-yl)-, methyl ester,(E)-	$C_9H_{12}O_2$	–	0.659	1.735	–
10.82	Phenol, 4-ethyl-2-methoxy-	$C_9H_{12}O_2$	2.055	2.571	1.770	2.167
11.037	4H-1-Benzopyan-4-one, 3-(3,4-dimethoxnyl)-6,7-dimethoxy	$C_{19}H_{18}O_6$	2.276	2.336	2.054	2.256
11.278	1H-Indene, 1-ethylidene-	$C_{11}H_{10}$	0.176	0.471	–	–
11.383	2-Methoxy-4-vinylphenol	$C_9H_{10}O_2$	5.549	9.316	5.659	7.814
11.918	Phenol, 2,6-dimethoxy-	$C_8H_{10}O_3$	1.376	2.841	1.515	1.715
11.987	Phenol, 2-methoxy-4-propyl-	$C_{10}H_{15}O_2$	–	0.835	–	–
13.209	Coumaran-3-one, 6-methoxy-2-[4-methoxybenzylidene]-	$C_{17}H_{14}O_4$	3.700	4.013	2.462	3.797
13.326	Biphenylene	$C_{12}H_8$	0.768	0.885	–	–
15.473	Benzoic acid, 2,4-bis[(trimethylsilyl)oxy]-, trimethylsilyi ester	$C_{16}H_{30}O_4Si_3$	2.493	2.346	2.071	2.487
17.811	1,6-Undecadiene, 2,6-dimethyl-, (E)-	$C_{13}H_{24}$	0.214	0.575	–	–

residence time. Reaction temperature showed similar positive impact on the bio-oil yield as residence time. The yields of bio-oil and volatile were ranged from 20.3 to 33.1% and 52.3 to 86.2%, respectively. The maximum bio-oil yield was obtained at the reaction temperature of 650 °C and residence time of 18 min while the maximum volatiles yield of 86.2% was obtained at 600 °C and 13 min.

3.3. *Determination of components in bio-oil by GC–MS*

To further understand the chemical reactions from microwave pyrolysis, GC–MS analysis was carried out to determine the composition of bio-oils. The bio-oil samples were prepared from the pyrolysis of PCG by microwave heating at 550 and 650 °C with different reaction times. Major chemicals in bio-oil products are shown in Table 2. The product peaks were assigned by automatic library search NIST98.

According to Ateş and Iskdag [23], the most important point at investigating the similarity of bio-oil as a fuel is the aliphatic compounds, i.e., alkanes and alkenes. Aliphatic compounds present in the bio-oils from PCG1 to PCG4 were 36.9%, 28.6%, 43.5% and 36.7%, respectively. The highest yield of alkanes, 6.0%, was found in PCG2 whereas the highest yield of alkenes, 7.5%, was found in PCG3. Aromatic hydrocarbons present in bio-oils were benzenes, phenols and naphthalene. Benzenes detected in bio-oils were from 6.3% (PCG4) to 12.8% (PCG2). Phenols derived from bio-oil have been looked at as a potential substitute for petroleum-based phenols [24], which are quite expensive. Phenols present in the bio-oil were from 25.3% (PCG1) to 39.8% (PCG2) depending on the pyrolysis conditions, which were made up of phenol, diphenols, methyl phenols, ethyl phenols, and methoxy phenols. Naphthalene was only detected in PCG2 (0.18%). The acetic acids were considered a main component in the liquid bio-oils from lignocelluloses biomass pyrolysis. In this study, carboxylic acids were determined in the oils as 2.8–4.6%, thus the bio-oils show low pH values (2.2–2.6). Nitrogen bases identified in the PCG bio-oil included 3-methyl-Pyridine, 1H-Imidazole-4-methanol, and 1-methyl-1H-1,2,4-Triazol-3-amine.

Fig. 3 shows the carbon number distributions for bio-oil samples prepared from the pyrolysis of PCG by microwave heating at 550 and 650 °C with different reaction times. The analysis of chromatograms and carbon number distribution showed that the concentration of compounds varies with different carbon numbers in the bio-oil samples. This result confirmed that the chemical composition of bio-oil was temperature and time dependent. It was noted that the range of carbon sizes run from C4 to C20, with the most prevalent sizes, C5, C6, C7, C8, C9, C11, C12, C14, C16, and C17.

3.4. *Kinetics evaluation*

PCG is mainly comprised of cellulose, hemicellulose, and lignin. PCG is a complex solid with the volatile content which is released during pyrolysis. Products of microwave pyrolysis were obtained in three fractions, bio-char, bio-oil, and syngas which proportions depended on the process conditions. Parameters like temperature

Table 3
Kinetic parameters for the microwave pyrolysis of PCG by Coats–Redfern method.

Raw materials	Reaction order	Activation energy E (kJ/mol)	Frequency factor A (s^{-1})	R^2
PCG	$n = 1$	3.10	2.26	0.42
	$n = 2$	13.3	1.82	0.91
	$n = 3$	12.1	1.53	0.89

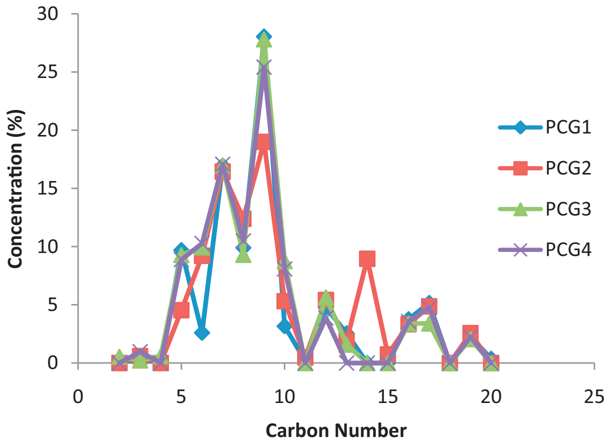

Fig. 3. Carbon number distribution of PCG bio-oils. PCG1: 550 °C and 8 min; PCG2: 650 °C and 8 min; PCG3: 550 °C and 18 min; and PCG4: 650 °C and 18 min.

or residence time influenced the products through the kinetics of the reaction; therefore knowledge of the kinetics is a key factor to predict product yields. Using data from the study, the kinetic parameters including the activation energy (E) and the frequency factor (A) were estimated and listed in Table 3. As can be seen in Table 3, a second-order reaction mechanism fits well the microwave pyrolysis of PCG with $R^2 = 0.91$. A constant heating rate was used with activation energy around 13.3 kJ/mol for PCG and a frequency factor of 1.82 s^{-1}. The model gives a good approximation of the temperature range where the reaction takes place at the constant heating rate of pyrolysis process. The good fit of the pyrolysis path is carried out depending on the temperature and time. Regarding to the evolution of the total volatiles with temperature and heating rate, the model is able to describe properly the experimental data.

4. Conclusions

In this research, microwave pyrolysis of PCG was performed under various conditions. Reaction temperature and residence time was observed to be significant factors affecting the production of bio-oils. The pyrolysis process variables included reaction temperature (529–671 °C) and residence time (5.9–20.1 min). The bio-oil yield was ranged from 20.3 to 33.1%. The maximum bio-oil yield, 33.1% was obtained at the reaction temperature of 650 °C and residence time of 18 min. Three linear models as a function of reaction temperature and time were established to predict the yield of bio-oil, syngas and bio-char. Second-order reaction kinetics was developed to model the pyrolysis of PCG. The results of GC–MS tests indicate that a series of chemical compounds were found in the bio-oils, including aliphatic compounds and aromatic hydrocarbons, which were related to the pyrolysis conditions. The range of carbon sizes run from C4 to C20, with the most prevalent sizes, C5, C6, C7, C8, C9, C11, C12, C14, C16, and C17.

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